

Final Report on

Transient Liquid Phase Bonding of Ferritic Oxide Dispersion Strengthened Alloys

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1 Executive Summary

This report presents work on the bonding of the ferritic ODS alloys MA956 and PM2000, principally by transient liquid phase (TLP) bonding, although diffusion bonding is also touched on briefly. In this work it is shown that, via the use of thin ($\leq 1 \mu\text{m}$) sputtered boron interlayers, high quality TLP bonds can be obtained. However, the orientation of the faying surfaces is of vital importance in bond quality. Results from an initial study of diffusion bonding are also promising.

2 Background

Ferritic oxide dispersion strengthened (ODS) alloys are attractive candidates for fuel can applications in the nuclear industry. However, deployment of ferritic ODS alloys in these types of applications requires the availability of a reliable joining technology for bonding end-caps onto the cans. Candidate joining technologies must be capable of producing reliable high-temperature performance in the bonded component.

ODS alloys present a significant challenge for joining, as it is vital to avoid disrupting the distribution of the oxide dispersion. Hence, conventional joining techniques, such as fusion welding are not particularly attractive for ODS alloys. The present work therefore focuses on transient liquid phase (TLP) bonding. A limited amount of work on diffusion bonding (DB) is included for comparison.

3 The Transient Liquid Phase Bonding Process

TLP bonding (see [1] for the first formal description of this process, which prior to that time occurred serendipitously) is derived from high-temperature fluxless vacuum brazing. In common with brazing, an interlayer is placed between the faying surfaces, such that a liquid is produced below the solidus temperature of the substrates. Most commonly, liquid formation depends upon an eutectic reaction, either within the interlayer, or between the interlayer and the substrates. Given a sufficiently protracted hold at the bonding temperature, diffusion of interlayer constituents into the substrate and/or vice-versa induces compositional changes that result in isothermal solidification at the bonding temperature. After a further solid-state homogenization treatment, bonds with chemistries, microstructures and hence high-temperature mechanical properties similar to those of the bulk substrates may be produced. One of the present authors has undertaken a detailed review of the TLP bonding literature and this may be found in reference [2].

There has been a limited amount of prior work on TLP bonding of ODS alloys and problems were found with producing the desired microstructure in the bonds. For example, Markham [3] observed uncontrolled boron-induced recrystallization when bonding nickel-base ODS alloys with relatively thick melt-spun boron-containing foils (typically such foils are in the 25 – 50 μm range). Khan and Wallach [4, 5] had rather greater success when using thin-film sputtered interlayers to bond ferritic ODS alloys, especially when bonding from the fine-grained condition and conducting a secondary

recrystallization treatment after bonding, but were unable to induce microstructural continuity across the bond-line.

4 Selection of TLP Bonding Compositional and Process Parameters

For the present work, the authors followed Khan and Wallach (see above) in using thin sputtered interlayers to minimize dissolution of the substrates and hence disruption of the dispersoid distribution. Again following Khan and Wallach, bonding was mostly conducted in the fine grain (FG), unrecrystallized condition (with some work performed on substrates in the coarse grained (CG), recrystallized condition for comparison), so as to allow at least some opportunity for the development of a uniform, recrystallized microstructure after bonding. However, two crucial changes were made for the present study. Firstly, the work examined the effect of bond orientation (see Figure 1). All previous work found in the literature invariably had the bond-line perpendicular to the working direction, with the result that all of the dispersoid stringers were cut by the bond (bond type “D” in Figure 1). Secondly, a significant applied load (1 – 5 MPa) was used to extrude out most of the liquid and hence minimize damage to the dispersoid distribution induced by excessive dissolution of the substrates.

Two ferritic ODS alloys, namely MA956 and PM2000 were used as the substrate materials in this work. Pure boron interlayers were produced by electron beam physical vapor deposition (EBPVD) to produce thicknesses of 250 nm, 500 nm and 1 μm . A commercial 25 μm Fe – 16 at. % B – 5 at. % Si melt spun foil (donated by Metglas Inc.) was used for comparison.

Bonding was conducted under a 10^{-3} Pa vacuum, using a Gleeble 1500 thermomechanical processing system, at a temperature of 1,250 °C, selected to be well clear of the Fe – B binary eutectic temperature. An applied stress in the range 1 – 5 MPa was employed. The typical heating rate utilized was 8 – 10 °C s⁻¹, which was found to be sufficiently high to prevent excessive diffusion of boron into the substrates during heating to the bonding temperature. Various bonding times (typically less than 5 minutes) were examined.

Post-bond heat treatment (PBHT) was performed both to induce recrystallization and to continue diffusion of boron into the substrates. Various PBHT times were investigated, at temperatures of 1,300 and 1,385 °C for the MA956 and PM2000 materials, respectively. A limited amount of work was also performed at lower PBHT temperatures.

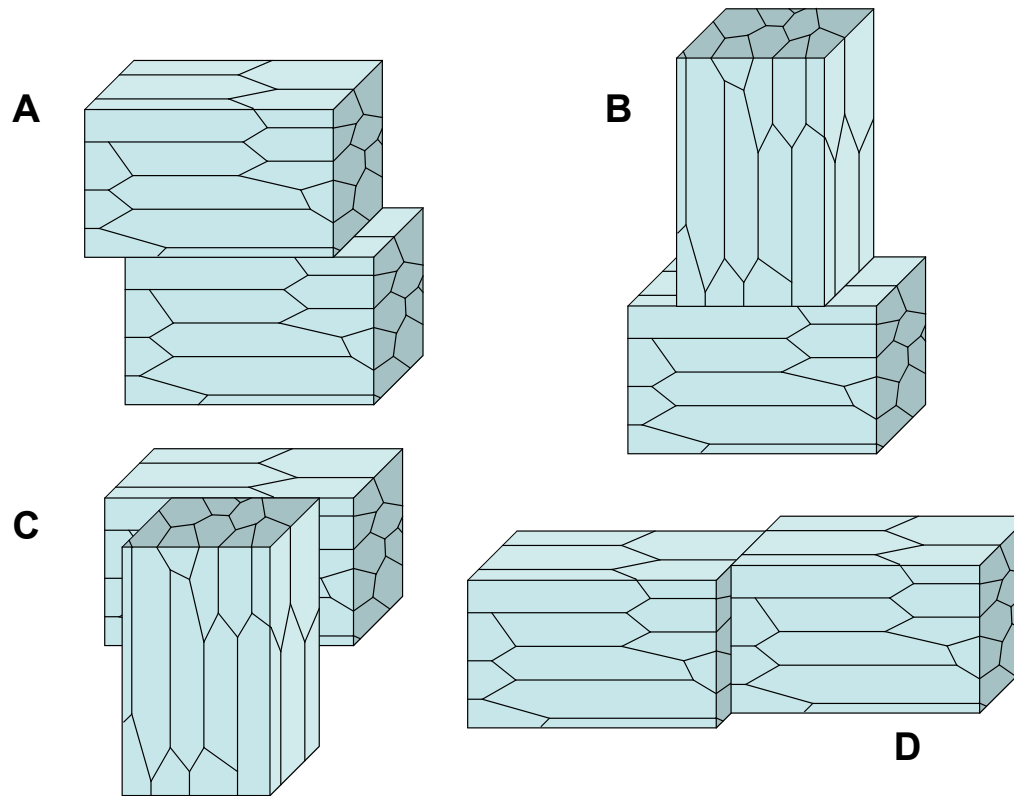


Figure 1. Bond geometries investigated (note the laps shown are for illustration purposes and butt joints have been employed):

- A. longitudinal substrate bonded to a parallel longitudinal substrate, with the bond-line parallel to the working direction;
- B. longitudinal substrate bonded to a perpendicular longitudinal substrate, with a mixed bond-line orientation;
- C. longitudinal substrate bonded to a transverse substrate, with a mixed bond-line orientation;
- D. longitudinal substrate bonded to a parallel longitudinal substrate, with the bond-line perpendicular to the working direction.

5 Outcomes from the Transient Liquid Phase Bonding Work

Little difficulty was encountered in controlling boride formation within the joints and from that standpoint, all of the work conducted was successful. However, bond-line orientation had a very large effect on the ability to control secondary recrystallization in the vicinity of the bond-line. The best results were obtained bonding using the “A” orientation in Figure 1, since this avoided cutting multiple dispersoid stringers. Notice that there is almost complete microstructural continuity for the MA 956 bond shown in Figure 2. In such bonds, uncontrolled secondary recrystallization only occurred when excessive liquid was left in the bond cavity, rather than extruding this out. Contrast this with the worst case, namely the “D” orientation in Figure 1, for which every single dispersoid stringer is cut by the bond-line. In the case of the D orientation, uncontrolled recrystallization invariably occurred and Figure 3 shows the best results obtained, for a D orientation bond, for which undesired fine grains are still clearly evident.

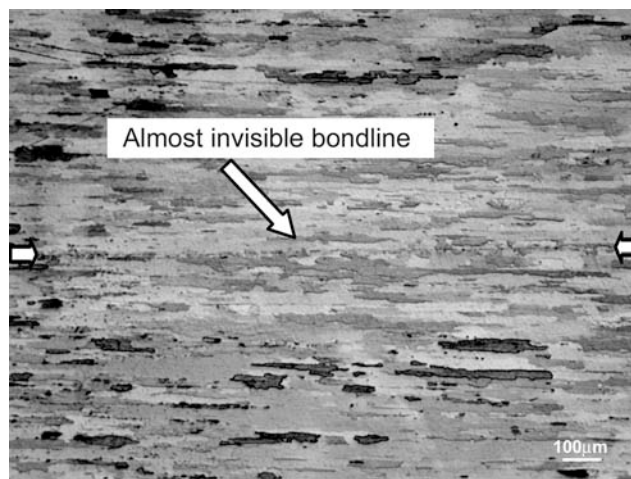


Figure 2. MA956 fine grain bond, made in the “A” orientation, as defined in Figure 1. Bonding was achieved, using a 1 μm boron interlayer, by holding for 120 s at 1250 $^{\circ}\text{C}$, followed by PBHT for 1 hr at 1300 $^{\circ}\text{C}$. Small arrows show the bondline. Note the almost complete absence of fine grains at the bondline.

Similar results were achieved with PM2000 bonds and indeed it was found possible to promote growth of coarse columnar grains right across the bond, as can be seen in Figure 4. Compared with the stark effect of substrate orientation, the exact thickness of the interlayer was found to be less critical within the range examined (250 nm to 1 μm). Likewise, so long as a compressive stress of at least 1 MPa was applied, increasing this up to 5 MPa had little effect. Given the use of such thin interlayers (the liquid thickness formed by reaction with the substrates is of course much greater than that of the as-deposited boron, but these are still very narrow gap bonds), careful surface preparation for parallelism and precise fixturing were found to be critical, which may cause scale-up issues.

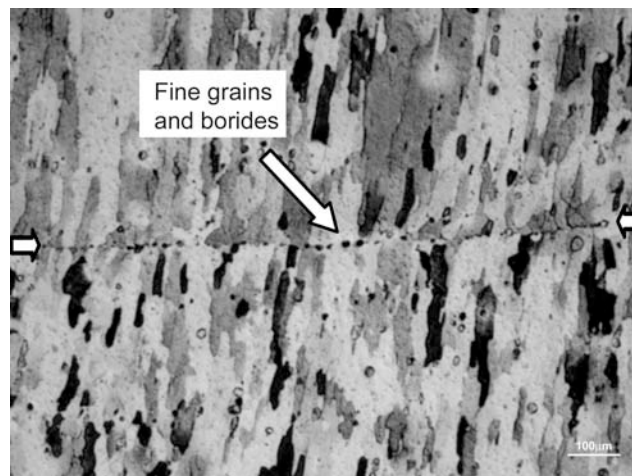


Figure 3. MA956 fine grain bond, made in the “D” orientation, as defined in Figure 1. Bonding was achieved, using a 1 μm boron interlayer, by holding for 256 s at 1250 °C, followed by PBHT for 1 hr 1250 °C + 1 hr at 1300 °C. Small arrows show the bondline. Notice the bondline is much more distinct than for the “A” orientation bond in Figure 2.

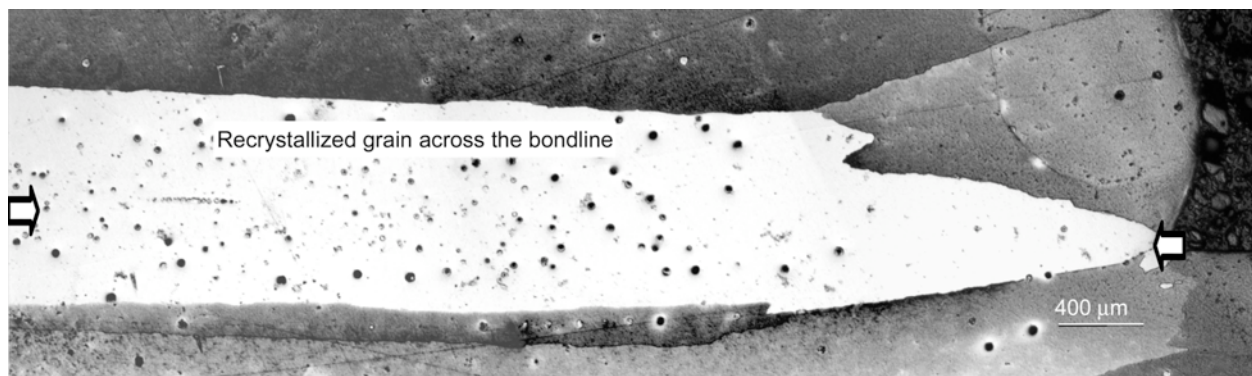


Figure 4. PM2000 fine grain bond, made in the “A” orientation, as defined in Figure 1. Bonding was achieved, using a 250 nm boron interlayer, by holding for 30 s at 1250 °C, followed by PBHT for 2 hr at 1385 °C. Small arrows show the bondline. Notice that recrystallization has occurred right across the bondline.

Problems were encountered in the PM2000 samples with the formation of porosity in some of the bonds. Whenever porosity was observed at the bond-line, this also occurred in the bulk substrates (although sometimes to a lesser extent) and was generally found to be associated with the use of material from specific regions of the PM2000 bar stock. Gas evolution is a generic problem with ODS alloys (see e.g.[6]). Despite extended soaking below the recrystallization temperature (e.g. 1,100°C for 70 hours), based on work by Chen *et al.* [7] and experimenting with a variety of heating ramps and holding temperatures, it was not found possible to completely eliminate the pores from all samples and greater control over the feedstock appears to be needed.

Unlike the bonds made using thin ($\leq 1\ \mu\text{m}$) boron interlayers, it was found to be impracticable to control secondary recrystallization of bonds prepared using the $25\ \mu\text{m}$ Fe – Si – B interlayer, as can be seen in Figure 5, although it did prove surprisingly easy to control boride formation in these bonds.

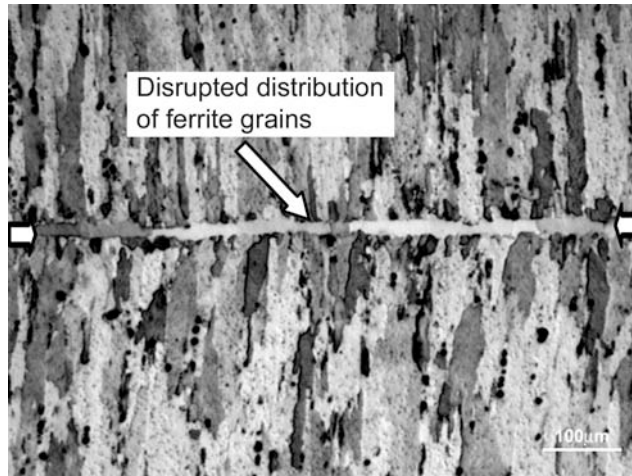


Figure 5. MA956 fine grain bond, made in the “D” orientation, as defined in Figure 1. Bonding was achieved using a $25\ \mu\text{m}$ Fe–Si–B interlayer, by holding for 150 s at $1,190\ ^\circ\text{C}$, followed by PBHT for 1 hr at $1,300\ ^\circ\text{C}$. Small arrows show the bondline. Notice that the microstructure of the bondline is quite unlike that of the substrates.

Results of room-temperature mechanical testing were fairly satisfactory, with bonds showing between 70 – 80 % of the strength of the bulk substrate after recrystallization treatment. Creep rupture testing commenced only late in the project, due to delays induced by attempts to address the porosity problem and results were not obtained for the TLP bonds.

6 Diffusion Bonding

The primary focus of this project was TLP bonding. However, it was also found possible to diffusion bond MA956 and PM2000. This is surprising given the likely faying surface oxide layers. Even more remarkable was the observation that diffusion bonding could be achieved under very low applied stresses ($\sim 5\ \text{MPa}$).

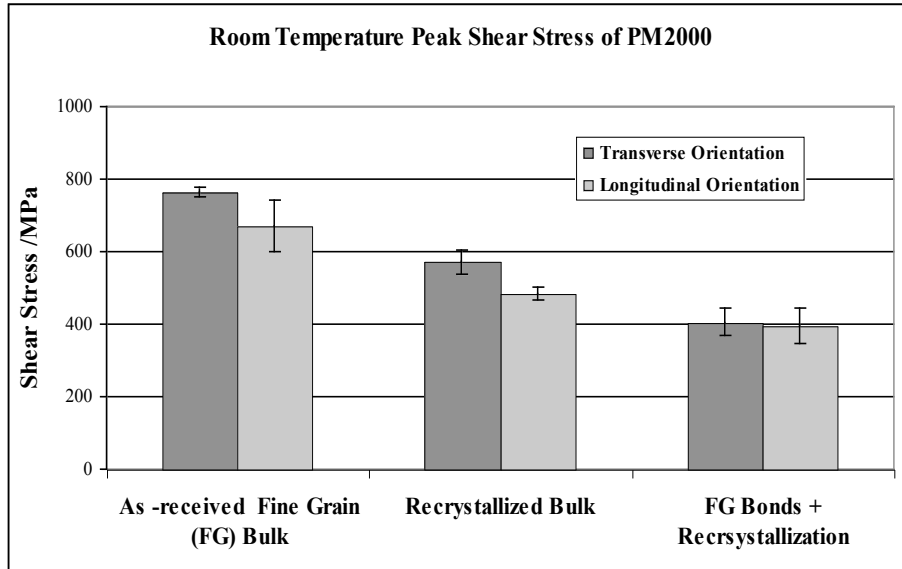


Figure 6. Room temperature shear tests of PM2000 in the as-received fine grain condition, recrystallized condition and bonded + recrystallized condition in both transverse (D) and longitudinal (A) orientations. Note the error bars represent standard deviation of the test results.

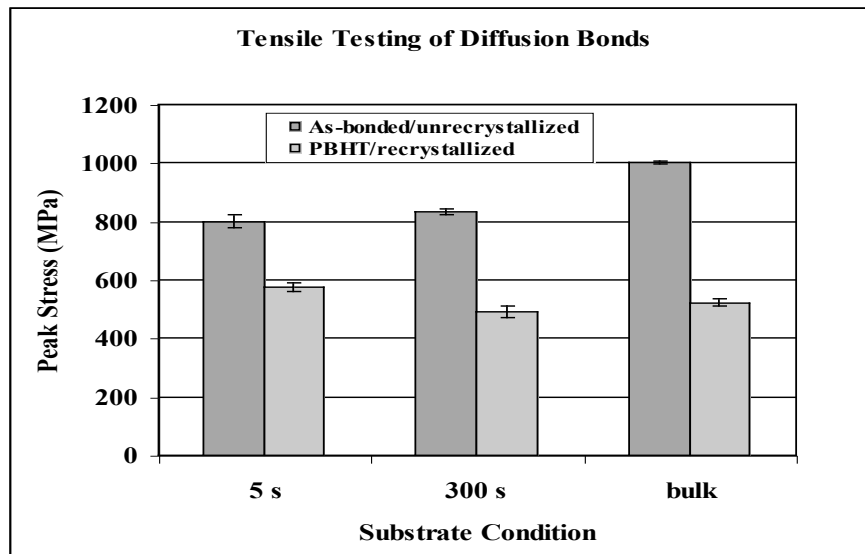


Figure 7. Room temperature tensile tests of PM2000 material, 5 s bond, 300 s bond, both at 1,250 °C and bulk in the unrecrystallized (■) and recrystallized (□) conditions. Note the error bars represent the standard deviation of the test results.

7 Conclusions

From the study of the joining of the ferritic ODS alloys MA956 and PM2000 described in this report, the following conclusions were drawn:

- ❑ The orientation of the faying surfaces has a very large effect on the ability to control secondary recrystallization during TLP bonding. Bonds prepared with a longitudinal – longitudinal faying surface orientation showed microstructural continuity across the bond-line.
- ❑ The use of thin ($\leq 1 \mu\text{m}$) sputtered boron interlayers produced satisfactory results, when combined with an applied load (1 – 5 MPa) to extrude out excess liquid. Bonding with 25 μm Fe–Si–B foils was much less successful.
- ❑ The TLP bonds showed reasonable room-temperature strength (70 – 80 % of that of the bulk material, subjected to the bonding + PBHT thermal cycle).
- ❑ The generic problem with porosity, which arises with mechanically alloyed ODS alloys was observed in the present work and could not be entirely eliminated.
- ❑ Diffusion bonding may also be a viable process route for these materials.

8 References

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